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Full length article

Effects of trace Gd concentration on texture and mechanical properties of hot-rolled Mg−2Zn−*x*Gd sheets

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Abstract

The effects of Gd concentration (0.1, 0.3, 0.7 wt%) on the microstructure, texture and mechanical properties of rolled and annealed Mg−2Zn−*x*Gd sheets have been investigated aiming to develop low cost and high ductile Mg−Zn−Gd sheets. Dynamic recrystallization, static recrystallization and grain growth during hot rolling process and annealing process were delayed with increase of Gd concentration, leading to fine grain microstructure. The rolled 0.1 wt% Gd sheet showed strong basal texture which remained stable after annealing process and exhibited medium elongation of about 25%. In contrast, the rolled 0.3 wt% Gd sheet had weak basal texture which transformed to non-basal texture with double peaks tilted about ±48° to the transverse direction due to the static recrystallization during annealing process. Consequently, the annealed sheets exhibited higher elongation of 40% along the rolling direction and 50% along the transverse direction due to the existence of non-basal texture. It is suggested that the minimum effective concentration for texture randomization in the Mg−2Zn−*x*Gd alloy is about 0.3 wt%.

Keywords: Magnesium alloys; Rolling sheet; Texture; Ductility

1. Introduction

Magnesium alloy sheets have attracted great attention due to their more and more applications in transportation and aerospace aiming to reduce weight and increase fuel efficiency. However, wrought magnesium sheets usually show low ductility and poor formability at ambient temperature due to their strong basal texture [1−3].

It has been found that texture of AZ31 alloys could be modified by using special processing technologies, such as equal channel angular rolling [4,5] and differential speed rolling [6]. Furthermore, the addition of trace rare earth element (RE) to magnesium proved to be an effective way to weaken or randomize their basal texture without using the above mentioned processing technologies. More encouragingly, weakened texture is of benefit to improve the ductility and formability of magnesium alloys. For example, N. Stanford and M.R. Barnett [7] found that extruded Mg−RE binary alloys showed weaker extrusion texture with a component with <11−21> parallel to the extrusion direction inferred as "rare earth texture". Moreover, higher elongation (about 20%) was observed in the extruded Mg−La and Mg−Gd alloys compared with the Mg−0.1 wt% Ca, Mg−0.99 wt% Al and Mg−1.87 wt% Sn alloys with the same grain size level. R. Cottam et al. [8] and B.L. Wu et al. [9] studied the influences and mechanisms of yttrium addition on the texture of binary Mg−Y alloys and it was also concluded that yttrium addition could weaken magnesium texture and enhance the ductility of magnesium alloys.

Currently, although the texture randomizing mechanism of RE on magnesium alloys remains unclear, the development of high ductile wrought magnesium sheets which shows promising

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for commercialization has been addressed by a number of authors [10−13], while the binary alloys has little industrial application prospects. For example, Y. Chino et al. [14] and J. Bohlen et al. [15] developed rolled Mg−Zn−Y, ZE10, ZEK100 and ZEK410 sheets exhibiting random texture with basal poles tilting about 35° from the normal direction (ND) towards the transverse direction (TD), high elongation of 30% along the TD and good stretch formability. The present author [11] have also developed a series of high ductile Mg−x(x = 1,2)%Zn−0.8%Gd sheets showing non-basal texture with basal poles tilting ±45° towards the TD and a maximum elongation of 45% along the TD. These sheets exhibited excellent formability at room temperature due to the high uniform elongation, high strain hardening rate and lower anisotropy. However, the high cost of these alloys due to the addition of costly RE and Zr elements restricts the practical utilization in industry. Therefore, reducing the cost of high ductile magnesium sheets becomes one of the most critical issues to promote the industrialization of high ductile magnesium sheets. Decreasing RE concentration seems to be a simple, fast and effective way. N. Stanford et al. [16] pointed out that the minimum concentration required to produce “rare earth texture” in Mg−RE alloys is 0.38 wt%Gd, 0.18 wt%La and 0.24 wt%Ce, respectively.

Thus, based on the previous researches, this paper investigated the effects of Gd concentration on the microstructure, texture and mechanical properties of rolled and annealed Mg−2.0Zn−xGd alloys sheets aiming to ascertain the minimum effective Gd concentration for randomizing texture and to develop low cost, high ductile magnesium sheets which would be promising for industry production.

2. Experimental method

The compositions of Mg−Zn−Gd alloys and their designations in this study are listed in Table 1. The cast billets were prepared with high purity 99.9% Mg, 99.9% Zn and 99.95% Gd by resistance melting under the protection of a mixed gas atmosphere of SF_6 (1 vol.%) and CO_2 (99%). The samples for hot rolling were cut from the ingot and homogenized at 450 °C for 10 h and then quenched into hot water. The rolling sample with an initial thickness of 15 mm was rolled to sheet with a final thickness of 1.7 mm after 6 passes without any surface or edge crack. During the first 3 passes, the reduction per pass is about 25% and the samples were reheated to 420 °C and held for 10 min after each pass. The reduction per pass increased to 35% and the reheating temperature decreased to 300 °C during the last 3 passes. The rolled sheets were annealed at 250, 275, 300, 325, 350 °C for 1 h, respectively.

Table 1
The analyzed chemical composition of the investigated alloys (wt.%).

Alloys	Zn	Gd	Mg
Mg−2Zn−0.1Gd	1.96	0.11	Balance
Mg−2Zn−0.3Gd	1.96	0.27	Balance
Mg−2Zn−0.7Gd	2.01	0.74	Balance

Microstructure observations were conducted on optical microscope, Philips XL30 ESEM-FEG/EDAX scanning electron microscope. The pole figures (0002), (10−11) and (11−20) were measured up to a tilt angle of 70° using the Schultz reflection method in the centre of the RD-TD plane in the rolled sample. Defocusing corrections were made using experimentally determined defocusing curves from random powder samples. Specimens with gauge length of 25 mm, width of 6 mm and thickness of 1.7 mm were cut from the rolled sample along the TD and the RD. Tensile tests were conducted at an initial strain rate of 10^{-3} s^{-1} at room temperature.

3. Results

3.1. Microstructure and texture of the as-rolled sheets

Fig. 1 show the microstructures of as-rolled Mg−2Zn−0.1Gd, Mg−2Zn−0.3Gd and Mg−2Zn−0.7Gd, which are abbreviated to 0.1Gd, 0.3Gd and 0.7Gd, respectively. There are striking differences in the microstructures of the three rolled alloys sheets. The 0.1Gd and 0.3Gd sheets exhibited microstructures consisting of dynamic recrystallized grains and deformation twins. The fraction of recrystallized grains and twins in the rolled 0.3Gd sheet were comparatively smaller than in the 0.1Gd sheet. When the Gd concentration increased up to 0.7 wt%, deformed microstructures were observed and recrystallized grains and deformation twins were hardly seen. It seems that the dynamic recrystallized grain size and recrystallization area fraction decreases with the increasing of Gd concentration. It is inferred that the increasing of the Gd concentration could retard dynamic recrystallization and suppress deformation twining, and the retarding effects depend on the Gd concentration. The result is consistent with the fact that RE element could delay recrystallization due to the low diffusion rate in magnesium caused by their large atoms radius [17].

The textures of the as-rolled Mg−2Zn−xGd sheets are shown in Fig. 2. All the rolled sheets exhibited basal textures with basal poles of peak intensity parallel to the normal direction. However, two vital differences among their textures should be noted. The first one is that the distribution range of the basal texture is distinct. The basal texture distributed uniformly towards the RD and the TD in the 0.1Gd sheet and the tilting angle of the texture component with intensity of 1 is about ±30° towards RD and TD. In contrast, the basal texture distributed much broader towards the TD than the RD in the 0.3Gd and 0.7Gd sheets, and the tilting angle of the texture component with intensity of 1 was about ±30° and ±60° towards RD and TD, respectively. The second is that the peak intensities of the basal textures are in different level. The peak intensity of the 0.1Gd sheet was about 7.7 while those of the other two alloys were 5.0 and 4.1, respectively. In general, the 0.1Gd sheet showed strong basal texture similar to rolled AZ31, while the 0.3Gd and 0.7Gd sheets exhibited weak basal textures with wider distribution towards the TD that is similar to the texture in the RE-containing alloys [14,15,18].

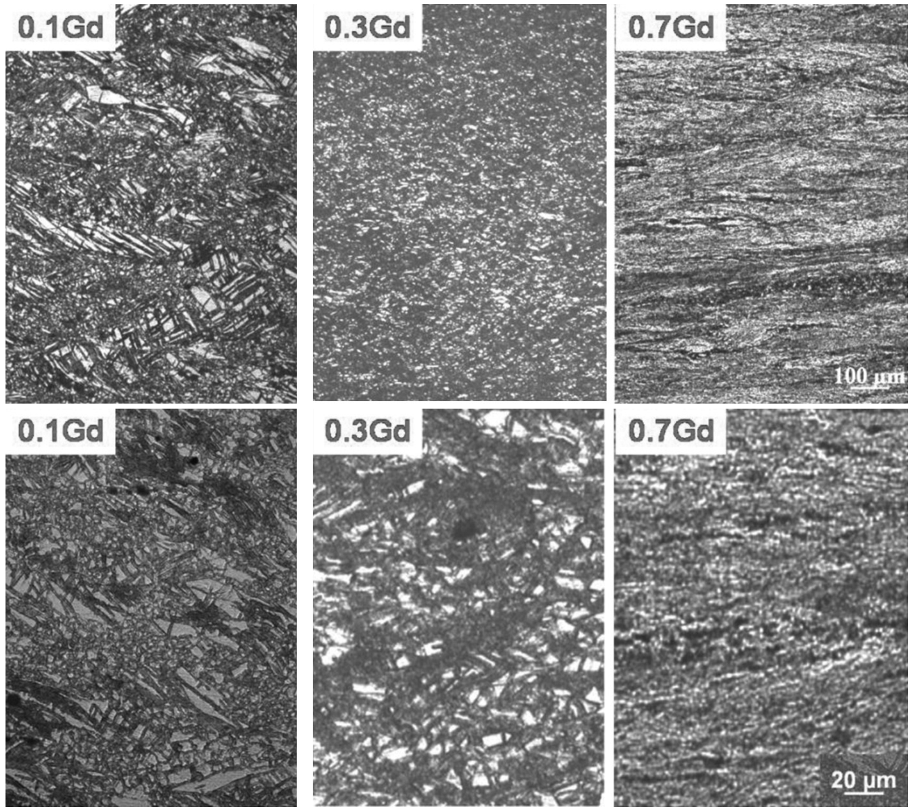

Fig. 1. Microstructures of the as-rolled Mg–2Zn–*x*Gd sheets.

Therefore, it is believed that Gd concentration has great influences on microstructures as well as textures.

3.2. Mechanical properties of the as-rolled sheets

Fig. 3 shows the engineering stress–strain curves of the as-rolled Mg–2Zn–*x*Gd sheets along the RD and the TD, respectively. It can be seen the yield strength increases with the increasing of Gd concentration both along the RD and the TD. The 0.1Gd sheet showed elongation of about 25% and low strength of around 220 MPa. Besides, it exhibited isotropic mechanical properties including yielding strength and elongations along the RD and the TD. In comparison with that, the 0.3Gd and 0.7Gd sheets have obvious anisotropy, such as higher yield strength along the RD than the TD and larger elongation along the TD than the RD. The 0.7Gd rolled sheet has yield strength of about 325 MPa and 280 MPa along the RD and TD, respectively.

The differences of the mechanical properties are related to the distinct of microstructure as well as texture. The isotropy of the yield strength and elongations along the RD and the TD in the 0.1Gd sheet is attributed to the uniform distribution of basal texture towards the RD and TD and the uniform microstructure. Similarly, the mechanical properties anisotropy in the 0.3Gd and 0.7Gd sheets is caused by the broader distribution of basal texture towards the TD. Besides, the accumulated stress during rolling process resulting from the difficulty in dynamic recrystallization due to the high Gd concentration is also responsible for the anisotropy of mechanical properties.

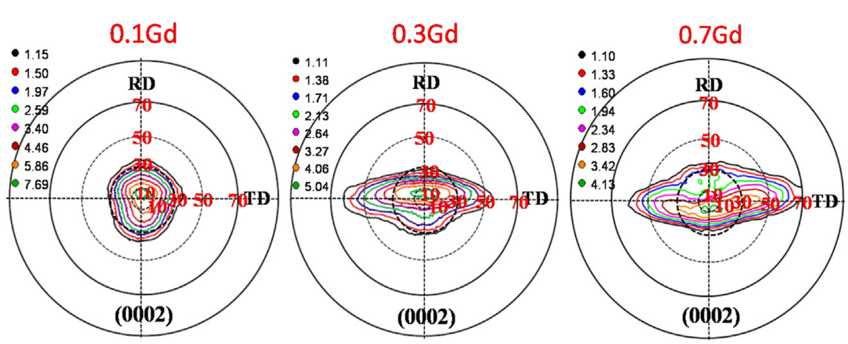

Fig. 2. (0002) pole figures of the as-rolled Mg–2Zn–*x*Gd sheets.

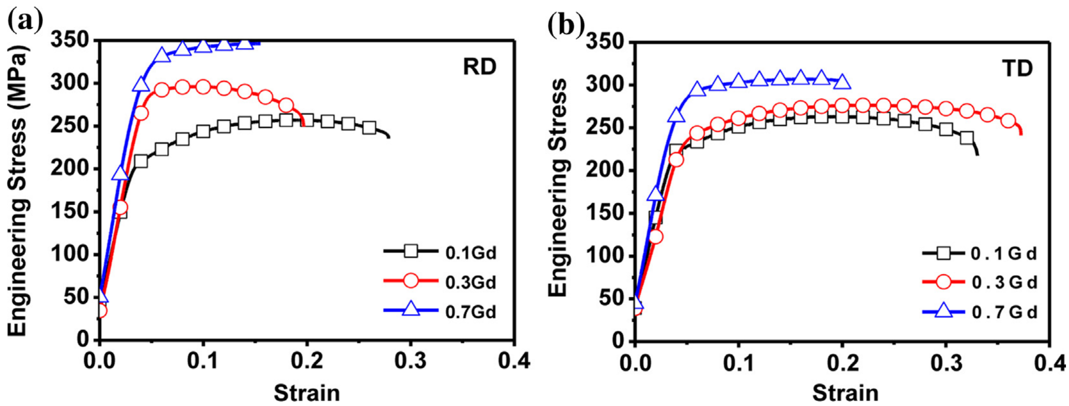

Fig. 3. Engineering stress–strain curves of the as-rolled Mg–2Zn–*x*Gd sheets along (a) the RD and (b) the TD, respectively.

3.3. Effects of annealing temperature on the microstructure and texture

The effects of annealing temperature on the microstructures of Mg–2Zn–*x*Gd sheets are shown in Fig. 4. The annealing temperature affects greatly the microstructures and grain size of the sheets with different Gd concentration. The 0.1Gd and 0.3Gd sheets annealed at 250 °C for 1 h showed a fully recrystallized microstructure indicating static recrystallization taking place during annealing process even at 250 °C, while 0.7Gd sheet annealed at 275 °C still had a deformed microstructure. It should be noted that although the 0.1Gd and 0.3Gd sheets had similar initial static recrystallization temperature, their microstructural evolution during annealing process at higher temperature were quite different. In the 0.1Gd sheet, the grain size increased dramatically and rapid grain growth took place when annealing temperature increased from 275 °C to 350 °C. In contrast, the grain size remained constant in the 0.3Gd sheet even the annealing temperature increased up to 350 °C.

The relationship between grain size and annealing temperature in the three alloys is shown in Fig. 5. The grain size increased dramatically from 12 μm to 78 μm with the increasing of annealing temperature from 250°C to 350 °C in the 0.1Gd sheet. However, in the 0.3Gd and 0.7Gd sheets, the grain size increased slowly from 9.5 μm to 17 um with increasing of annealing temperature. It is interesting to note that the grain size is much stable even at higher annealing temperature when Gd concentration increases only from 0.1 wt.% to 0.3 wt.%. Combining with the microstructural evolution during annealing process of the 0.1Gd and 0.3Gd sheets, it is indicated that the increasing of Gd concentration not only retards dynamic recrystallization, but also delays static recrystallization and grain growth.

Fig. 6 shows the textures of the Mg–2Zn–*x*Gd sheets annealed at 275, 300, 325 and 350 °C for 1 h, respectively. The annealed 0.1Gd sheet exhibited strong basal texture similar to the as-rolled state, and the basal texture remained stable with increasing annealing temperature. It seems that annealing process had little effect on texture type of the 0.1Gd sheet. However, the peak intensity of the texture increased dramatically from 7.69 to 32 with the increasing annealing temperature due to rapid grain growth. In contrast, in the 0.3Gd and 0.7Gd sheets the annealing process has vital influences on texture type. Non-basal textures were observed after annealing process, which were completely different from the basal texture of the as-rolled state but with almost the same peak intensity. However, some differences between the 0.3Gd and 0.7Gd annealed sheets were observed. The 0.3Gd sheet annealed at 275°C had non-basal texture with one peak tilted about 43° to the TD, which transformed to non-basal texture with asymmetrical double peaks tilted about 43° to the TD with the annealing temperature increasing to 350 °C. In contrast, the 0.7Gd sheet annealed at 275 °C showed non-basal texture with three peaks in which one peak was basal texture component and the other two were asymmetrical non-basal texture components with tilting angle of about ±40° to the TD, as shown in Fig. 6(c). The basal texture component disappeared and only the two asymmetrical non-basal texture components remained when the annealing temperature increased to 300 °C. The non-basal texture components were stable even the annealing temperature reaching to 350 °C and the tilting angle of the double peaks increased to about 50°, which was larger than that in the 0.3Gd annealed sheet.

Consequently, the Gd concentration affects not only the dynamic recrystallization texture but also the annealing texture. The basal texture in the 0.1Gd sheet remained stable during annealing process, while the basal texture changed from strong basal texture with broader distribution to the TD into non-basal texture with double peaks tilted to the TD after annealing process in the 0.3Gd and 0.7Gd sheets. Therefore, it is suggested the effects of annealing process on texture evolution is dominated by Gd concentration.

3.4. Mechanical properties of annealed sheets

Fig. 7 shows the engineering stress–strain curves of the annealed Mg–2Zn–*x*Gd sheets along the RD and the TD, respectively. There were striking differences in the mechanical properties between the annealed 0.1Gd sheet and the other two sheets. In the annealed 0.1Gd sheet, the elongation was around 25% both along the RD and the TD. Similar to the as-rolled

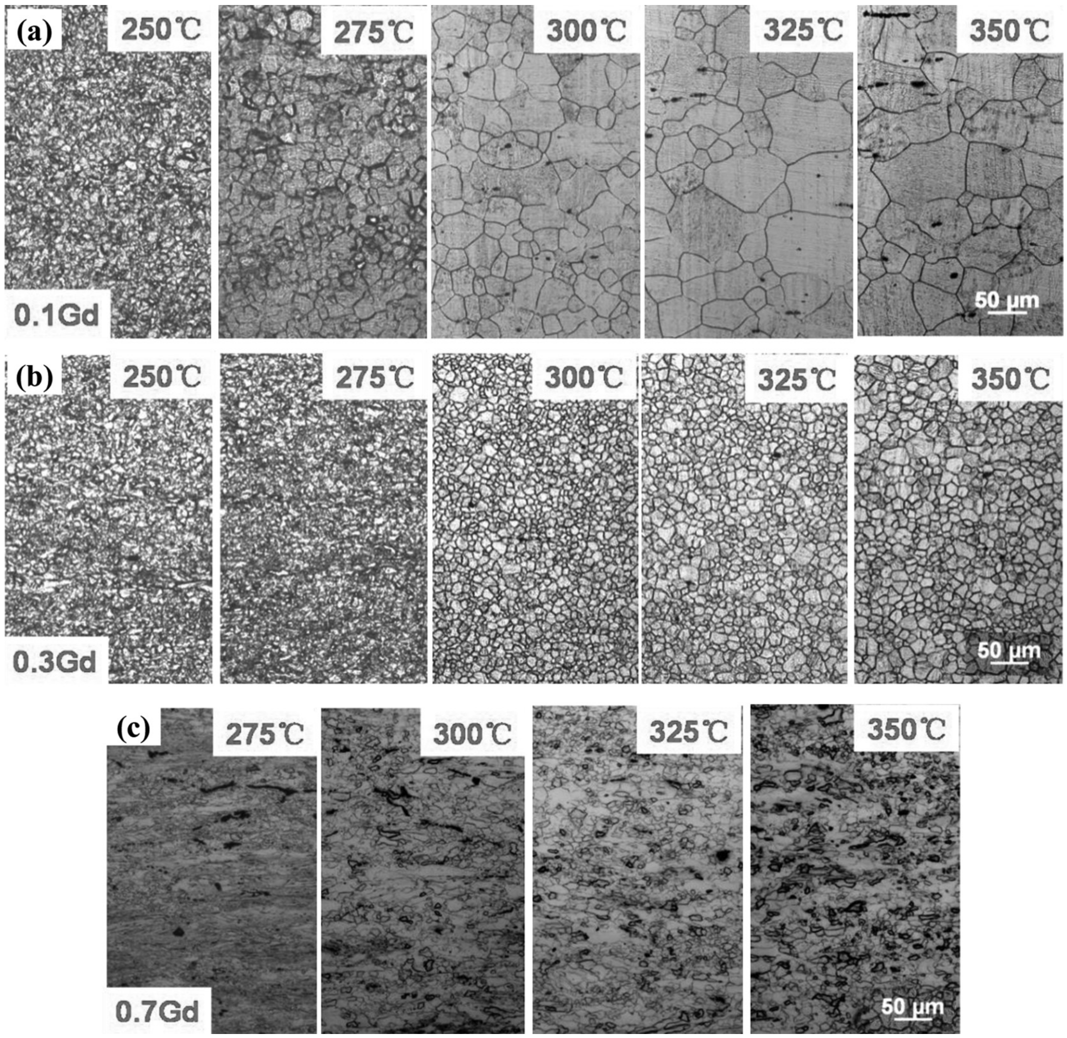

Fig. 4. The microstructures of the Mg–2Zn–xGd sheets annealed at 250, 275, 300, 325 and 350 °C for 1 h, respectively (a) 0.1Gd sheet, (b) 0.3Gd sheet and (c) 0.7Gd sheet.

state, the yield strength and elongation along the RD and the TD was almost the same. In addition, it seems annealing temperature had little influence on the mechanical properties. In contrast, annealing process had great effects on the yield

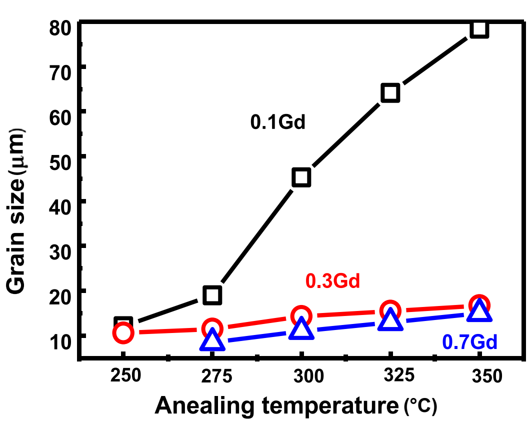

Fig. 5. The relationship between grain size and annealing temperature in the Mg–2Zn–xGd sheets.

strength and elongation of the annealed 0.3Gd and 0.7Gd sheets. After annealed at 275 °C, the elongation of 0.3Gd sheet increased up to 35% and 45% along the RD and the TD, respectively. Higher annealing temperature, i.e. 350 °C, is necessary for the 0.7Gd sheet to get the best elongations of 40% and 50% along the RD and the TD, respectively. It should be noted that yield strength is much lower along the TD than along the RD, while elongation was larger along the TD than along the RD.

4. Discussion

4.1. Effects of Gd concentration on the recrystallization behaviour and texture

Figs. 1 and 4 suggest that the Gd concentration has great influences on the rolling microstructure and the annealing microstructure. The influences manifest themselves mainly on the recrystallization behaviour, such as recrystallization temperature and grain growth. It is known that impurity and

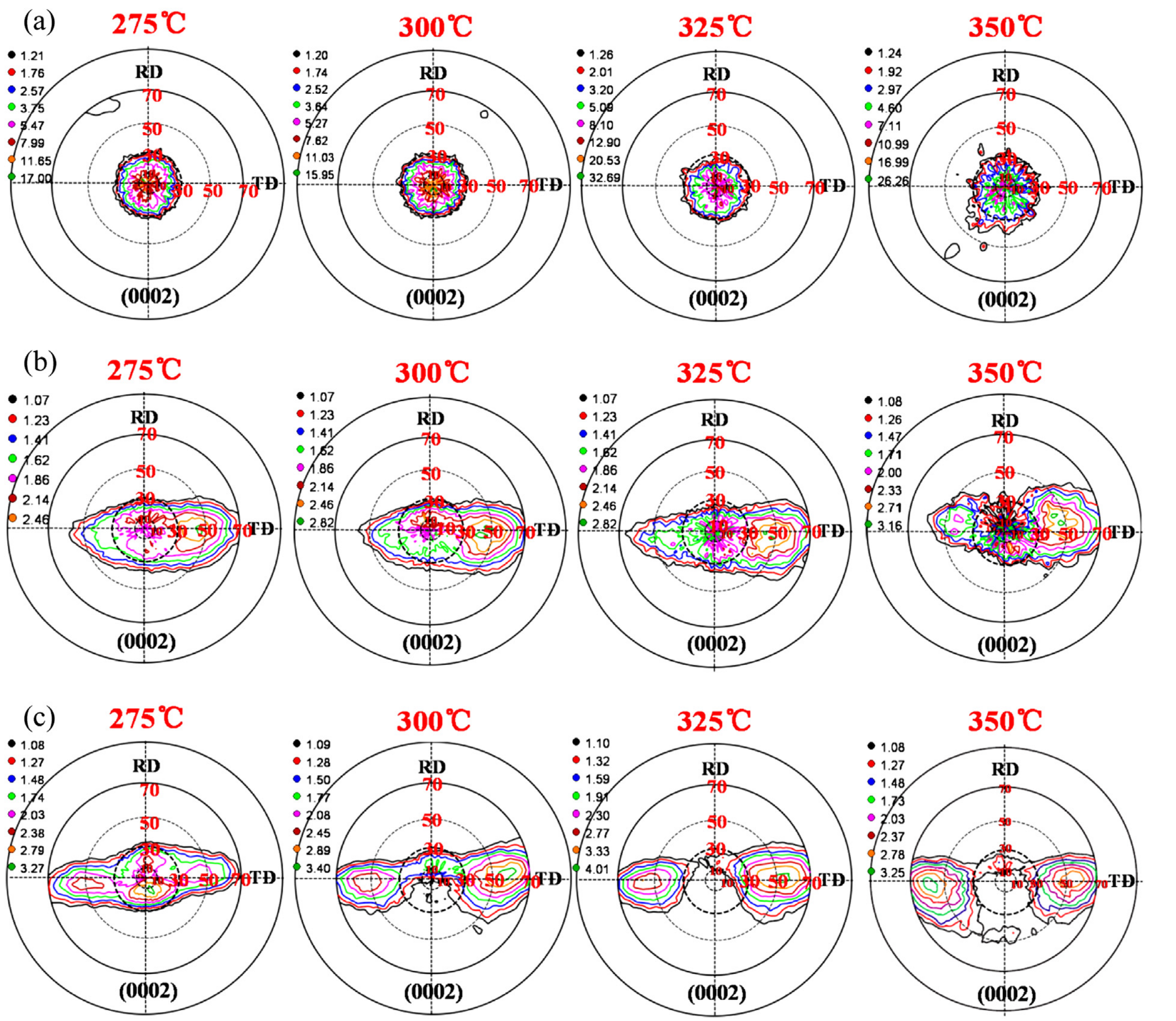

Fig. 6. (0002) pole figures of the Mg–2Zn–*x*Gd sheets annealed at 275, 300, 325 and 350 °C for 1 h, respectively (a) 0.1Gd sheet, (b) 0.3Gd sheet and (c) 0.7Gd sheet.

alloying elements suppress recrystallization and grain growth through delaying atom diffusing rate and new grain boundary migration. It was proposed that small addition of Mn or rare earth elements is effective for refining grain size and preventing grain coarsening during rolling and annealing process compared with Al and Zn elements [19]. The effects of alloying element concentration were not considered in those studies. The present research results show that alloying element concentration is also one key factor for grain refinement and dynamic recrystallization behaviour. In the ternary Mg–Zn–Gd alloys, the dynamic recrystallization and grain growth were delayed with the Gd concentration increasing during rolling and annealing process. Therefore, higher annealing temperature is required to obtain the uniform recrystallization microstructure in the Mg–2Zn–0.7Gd due to its higher Gd concentration.

The differences in the texture types among the Mg–Zn–*x*Gd sheets, for example, the basal texture in the annealed 0.1Gd sheet and the non-basal texture in the 0.3Gd and 0.7Gd sheets (Figs. 2 and 6), suggest that Gd concentration may be the key for the formation of non-basal texture. N. Stanford [16] has investigated the influences of RE element type (La, Gd, Y, Ce) and their concentration on the extruded texture of binary Mg–RE alloys, and pointed out that the minimum concentration required to produce “rare earth texture” is 0.38 wt%Gd, 0.18 wt%La and 0.24 wt%Ce, in binary Mg–RE extruded alloys, respectively.

Similar to the binary Mg–RE alloy, there is a critical value for the rare earth texture in the Mg–2Zn–*x*Gd alloys. It seems that the addition of 0.11Gd is invalid for the texture randomizing both in the binary Mg–Gd and the ternary Mg–2Zn–*x*Gd alloys. Although the addition of Zn would consume some Gd atoms due to the formation of ternary intermetallic particles, 0.27 wt%Gd is valid for the non-basal texture formation in the present ternary alloy. This transition of texture with increasing Gd concentration is consistent with the recrystallization behaviour and microstructure of the alloys in Figs. 1 and 4. Many mechanisms such as the *c*/*a* value,

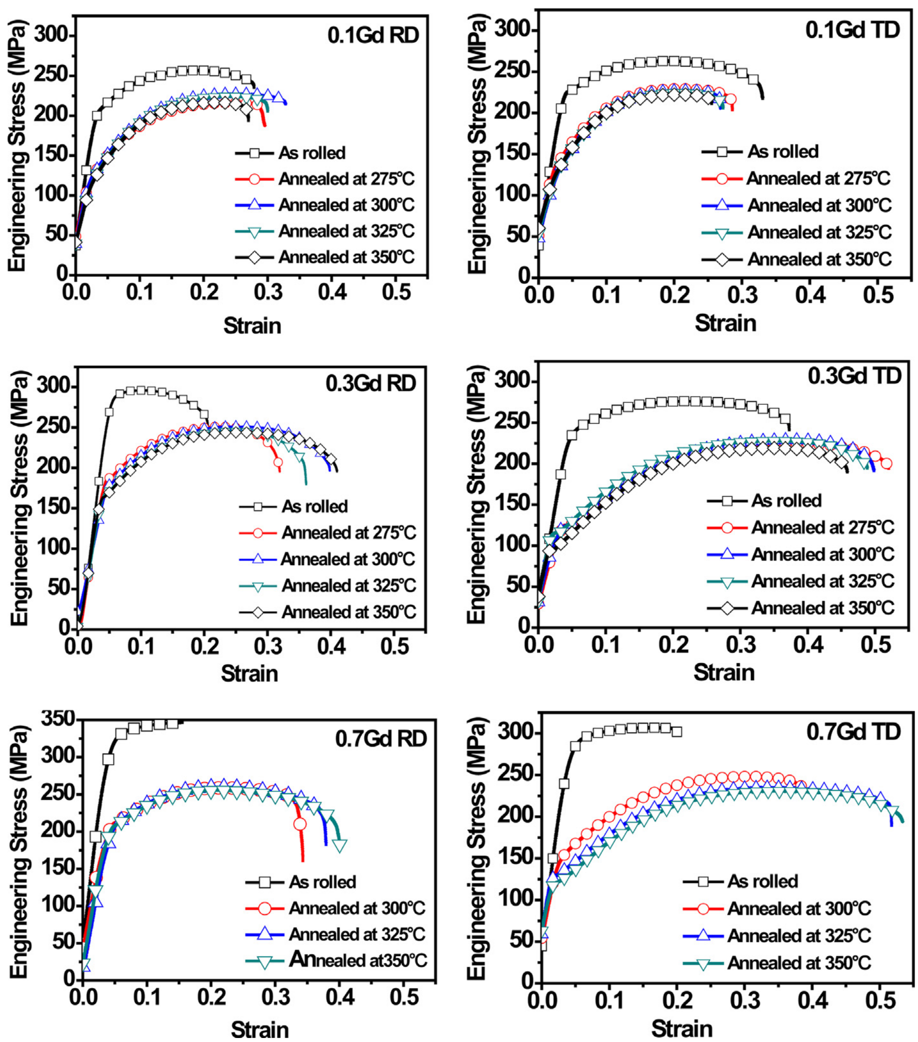

Fig. 7. Engineering stress–strain curves of the annealed Mg–2Zn–xGd sheets along the RD and the TD, respectively.

shear deformation and clustering or segregation of RE [20,21], have been proposed for the texture formation, although its essence remains unclear. Anyway, these mechanisms are related and sensitive to RE concentration, so there is a minimum effective concentration for these mechanisms as well as for the texture. Therefore, it is inferred that the increasing of Gd concentration from 0.11 wt% to 0.27 wt% cause some changes in crystal structure or microstructure which should be the key mechanism responsible for the texture randomizing effects and needs further investigation.

4.2. Influences of annealing process on the texture and mechanical properties

The texture evolution of AZ31 during annealing process has been studied, and it was proposed that annealing process have no function on the texture type. Initially, we were impressed that annealing process does not affect the texture of the present magnesium alloy based on the observations of AZ31. However, the texture in the rolled 0.3Gd and 0.7Gd sheets show basal texture with broader distribution to the TD, while the annealed sheets shows non-basal texture with double peaks tilted ±42° to the TD, indicating that the non-basal texture or texture randomizing are related to the annealing process, as shown in Fig. 6. Thus, it is proposed that annealing process could modify the texture of the hot rolled magnesium sheets.

However, it should be noted the transition of texture after annealing process needs two conditions. The first one is static recrystallization taking place during annealing process, because grain orientation does not change during normal grain growth in the completed dynamic recrystallized magnesium alloys. The second is that magnesium alloy should have texture randomizing-ability, otherwise, nothing would happen in texture except for some change in peak intensity as observed in AZ31 and the 0.1Gd.

The texture randomizing effect and mechanisms of RE addition on magnesium alloys have been observed and studied during rolling or extrusion process, in which dynamic recrystallization thoroughly took place. Some mechanisms, such as the continuous dynamic recrystallization in the shear bands, the low stacking fault energy, and the cluster solute atoms, have been proposed. For instance, it was proposed that the orientation of DRX grains were responsible for the weaken texture in Mg–Y alloy. The results here further confirm that the orientation rotation of recrystallized grain nucleation is responsible for the random texture no matter it is static recrystallization or DRX. Recrystallized grains orientation transition caused by the RE addition during recrystallization nucleation process should be key point for the texture random effect in the Mg alloy with RE addition, other than the deformation mechanism. Therefore, the dynamic recrystallization grains nucleation should be paid more attention for understanding the texture randomizing mechanism in magnesium alloys containing rare earth.

The huge differences in the ductility of the 0.1Gd sheet with basal texture and the 0.3Gd sheet with non-basal texture indicate that the texture type has great impact on the ductility. The poor ductility in magnesium with basal texture is related to the lack of sufficient deformation modes and the early shear failure due to the compression twining. The excellent ductility in the 0.3Gd and 0.7Gd alloys is attributed to the activation of basal slip and extension twinning, and non-basal slip during tensile deformation because of the non-basal texture.

5. Conclusions

The effects of Gd concentration and annealing process on the microstructure, texture and mechanical properties of rolled Mg–2Zn–*x*Gd alloys has been studied, and following conclusions can be drawn:

(1) Recrystallization and rapid grain growth is prone to take place during hot rolling process at lower rolling temperature in the 0.1Gd alloy, while it is much harder in the 0.3Gd and 0.7Gd alloys. The increase in Gd concentration could suppress and delay dynamic recrystallization during hot rolling process, static recrystallization and grain growth during annealing process.

(2) The Gd concentration has great influences on both the rolled and annealed texture. The rolled 0.1Gd sheet shows a basal texture, while the rolled 0.3Gd sheet shows a weak basal texture with broader distribution to the TD. After annealing process, the basal texture in the 0.1Gd retain unchanged, while the texture in the 0.3Gd transforms to non-basal texture with double peak tilted about ±48 °C to the TD. It is suggested that the effective minimum Gd concentration for texture randomizing in the Mg–2Zn–*x*Gd alloy is about 0.3 wt%.

(3) The non-basal texture observed in the annealed sheets containing 0.3Gd suggests that recrystallization be responsible for the texture random effect in magnesium alloy with RE addition no matter it is dynamic recrystallization or static recrystallization.

(4) The annealed sheets containing 0.1 wt%Gd exhibited elongation less than 25%, while the sheets containing 0.3 wt%Gd or more exhibited maximum elongation of 40% along the rolling direction and 50% along the transverse direction. The observed high elongation is attributed to the non-basal texture.

Acknowledgements

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